## **PCT**

# WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



INTERNATIONAL APPLICATION PUBLIS	HED U	JN	DER THE PATENT COOPERATI	ON TREATY (PCT)
(51) International Patent Classification 7:		(1	1) International Publication Number:	WO 00/04216
D04H 1/42, B01D 39/08, B03C 3/28	A1	(4	3) International Publication Date:	27 January 2000 (27.01.00)
(21) International Application Number: PCT/GB (22) International Filing Date: 24 June 1999 (			(81) Designated States: AE, AL, AM, BR, BY, CA, CH, CN, CU, C GD, GE, GH, GM, HR, HU, KP, KR, KZ, LC, LK, LR, LS,	Z, DE, DK, EE, ES, FI, GB, ID, IL, IN, IS, JP, KE, KG, LT, LU, LV, MD, MG, MK,
(30) Priority Data: 9815629.2 18 July 1998 (18.07.98)		B	MN, MW, MX, NO, NZ, PL, I SK, SL, TJ, TM, TR, TT, UA, ZW, ARIPO patent (GH, GM, UG, ZW), Eurasian patent (A RU, TJ, TM), European patent ES, FI, FR, GB, GR, IE, IT, I	UG, US, UZ, VN, YU, ZA, KE, LS, MW, SD, SL, SZ, M, AZ, BY, KG, KZ, MD, (AT, BE, CH, CY, DE, DK,
(71) Applicant (for all designated States except US): HEF MINERALS AND CHEMICALS LTD. [GB/GB ford Bridge, Kentmere, Nr Kendal, Cumbria LA8	]; Wate	er-	patent (BF, BJ, CF, CG, CI, C	
(72) Inventors; and (75) Inventors/Applicants (for US only): GORDO [GB/GB]; 1 Gallowsclough Road, Stalybridge, SK15 3QS (GB). COLLINGWOOD, Jeremy, [GB/GB]; 18 Hatlex Drive, Hest Bank, Lanca 6HA (GB). KENNAUGH, Martyn, Peter [GB/Greenwood, Kendal, Cumbria LA9 5ED (GB).	Cheshi Andre ster La /GB];	ire ew A2 18	Published With international search repo	rt.
(74) Agents: MCNEIGHT, David, Leslie et al.; Mcl Lawrence, Regent House, Heaton Lane, Stockport, SK4 IBS (GB).	Neight , Chesh	& ire		
(54) Title: FILTER MATERIALS AND METHODS FO	R THE	PR	ODUCTION THEREOF	
(57) Abstract		٠	•	
There is disclosed an electrostatically charged filter fibres or ii) PVC fibres or iii) a mixture of halogen free ac				s and: i) halogen free acrylic
			·	

### FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

						CI	Slovenia
AL	Albania	ES	Spain	LS	Lesotho	SI	Slovakia
AM	Armenia	FI	Finland	LT	Lithuania	SK	
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal Swaziland
ΑU	Australia	GA	Gabon	LV	Latvia	SZ	Chad
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	•
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav	TM	Turkmenistan
BF	Burkina Faso	GR	Greece		Republic of Macedonia	TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	18	Iceland	MW	Malawi	US	United States of America
CA	Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
СН	Switzerland	KG	Kyrgyzstan	NO	Norway	zw	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's	NZ	New Zealand		
CM	Cameroon	•-	Republic of Korea	PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
CU	Cuba	KZ	Kazakstan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Estonia	LR	Liberia	SG	Singapore		

# FILTER MATERIALS AND METHODS FOR THE PRODUCTION THEREOF

This invention relates to filter materials for filtering fluids and methods for the production thereof, with particular, but by no means exclusive, reference to filters for air filtration.

Air filter materials having permanent electrostatic charge are known. Such materials filter particles very efficiently due to electrostatic attractive forces between the material and the particles to be filtered. Applications include air filtration in dust helmets, respirators, suction cleaners and air conditioning systems.

Filter materials desirably possess a number of advantageous properties. Such materials should filter particles efficiently whilst providing low resistance to the flow of fluid through the filter material. In the case of air filtration, this means a low air pressure drop across the filter material. Furthermore, it is important that the filtering performance of the filtering material is maintained, or, at least, that deterioration in performance is minimised, for as long a period of time as possible. In this way, the working lifetime of the filter is extended. Furtherstill, it is important that, once the working lifetime of the filter is over, the filter can be disposed of safely.

European Patent EP 0 246 811 describes air filter material which exhibit good filtration efficiency and low resistance to air flow due to the open structure of charged fibres employed. The material comprises a blend of polyolefine fibres and halogen substituted polyolefine fibres. US Patent 5 470 485 describes air filter material comprising a blend of bi-component polypropylene/polyethylene fibres of the core/jacket or side-by-side type and halogen free polyacrylonitrile fibres. The use of the bi-component fibres is an essential element of US 5 470 485 so that stiff, mouldable non-

woven mats of filter material can be produced. It should be noted that in each case the filter material is overall electrically neutral, fibres in the materials of different type carrying charges of opposed polarities.

The present invention provides high quality filter material which exhibits some or all of the advantageous properties described above.

According to a first aspect of the invention there is provided an electrostatically charged filter material comprising a blend of polypropylene fibres and: i) halogen free acrylic fibres or ii) polyvinyl chloride (PVC) fibres or iii) a mixture of halogen free acrylic fibres and PVC fibres.

Such filter materials exhibits excellent filtering efficiency and low resistance to the flow of fluids through the material.

Preferably, the halogen free acrylic fibres are produced by dry spinning. In a dry spinning process, the acrylic polymer is formed into fibres by extrusion into an air stream. It has been found that the filter material comprising such fibres exhibit improved filtration efficiency and better long term charge stability.

The filter material may consist of a blend of polypropylene fibres and halogen free acrylic fibres. In contrast to the filter material of EP 0 246 811, such material may be safely disposed of by incineration, since dioxin compounds are not produced.

The filter material may consist of a blend of polypropylene fibres and PVC fibres. Such material exhibits excellent long term charge stability, leading to excellent filter performance over an extended time period.

The filter material may consist of a blend of polypropylene fibres, halogen free acrylic fibres, and PVC fibres. The presence of the PVC fibres in the blend provides enhanced performance over an extended time period.

The halogen free acrylic fibres may comprise at least 40%, preferably at least 50%, more preferably at least 60%, most preferably at least 85% acrylonitrile.

According to a second aspect of the invention there is provided a method for producing an electrostatically charged filter material comprising the step of blending together polypropylene fibres and: i) halogen free acrylic fibres or iii) PVC fibres or iii) a mixture of halogen free acrylic fibres and PVC fibres.

The performance of the filter material is considerably affected by the present of lubricants and anti-static agents (collectively known as a "spin finish") on the surface of the fibres. In practise, polymer fibres produced commercially have spin finishes, and thus it is necessary to substantially remove lubricants and anti-static agents from the fibres before, during or after the blending of said fibres.

The method may comprise the step of carding the blend of fibres so as to form a non-woven fabric. The non-woven fabric may be needled.

The halogen free acrylic fibres may be produced by dry spinning.

Polypropylene fibres may be blended with halogen free acrylic fibres but not with PVC fibres.

Polypropylene fibres may be blended PVC fibres but not with halogen free acrylic fibres.

- 4 -

Polypropylene fibres may be blended with halogen free acrylic fibres and PVC fibres.

Filter materials and methods for the product thereof will now be described with reference to the accompanying Figure, which shows penetration characteristics for two filter materials as a function of time.

The invention comprises a filter material comprising a blend of polypropylene fibres and: i) halogen free acrylic fibres or ii) PVC fibres or iii) a mixture of halogen free acrylic fibres and PVC fibres.

Filter material corresponding to each of options i), ii) and iii) was produced by a method described in more detail below. Filter material made from blends of polypropylene/halogen free acrylic and polypropylene/PVC were produced with blend ratios (by weight) of 50:50. Additionally, filter material was made from a blend of polypropylene/halogen free acrylic/PVC with a blend ratio of 50:25:25. However, it should be appreciated that the invention is not limited in this respect : different blend ratios may be employed. In general, a blend ratio of polypropylene/[acrylic + PVC] in the range of 70:30 to 30:70 is acceptable. There is economic advantage in using blend ratios in which the polypropylene component predominates, since this is the cheaper material. The ratio of halogen free acrylic to PVC is fully variable across the range 100:0 (all acrylic, corresponding to option i)) to 0:100 (all PVC, corresponding to option However, the term "polypropylene fibres" is understood not to include biii). side-by-side type core/jacket or the fibres, such as component polypropylene/polpropylene fibres described in US 5 470 485, which give rise to filter material having different physical properties.

The present invention recognises that the cleanliness of the fibres is of great importance if good filtering performance is to be obtained. In particular, there appears to be a relationship between the surface characteristics of the fibres used and long term performance of the filtering material. Fibres having surfaces that contain significant cracks and fissures generally produce inferior filtering material. It is likely that this is due, at least in part, to the difficulty of removing spin finish (and, possibly, water) from the cracks and fissures. Thus, it is preferable to utilise halogen free acrylic fibres which have been produced by dry spinning, in which the polymer is formed into fibres by extrusion into an air stream. Scanning electron microscopy has confirmed that fibres produced thereby are smoother and have less deformations than fibres produced by a wet spinning process, in which polymer solution is extruded into a coagulation bath. In the examples described herein, dry spun acrylic was used.

Filter material was produced generally according to the method described in EP 0 246 811. It should be noted that, in EP 0 246 811, removal of lubricants and anti-static agents is accomplished before blending of said fibres. The present examples, this removal step is performed <u>after</u> the step of blending the fibres. Both approaches are within the scope of the invention. The removal of lubricants and anti-static agents can be performed by a textile scouring process, such as detergent, alkali or solvent scouring. It is preferred to use a detergent scouring process, followed by rinsing and drying of the fibres.

The blend is then carded, a process which i) forms a non-woven fabric and ii) enables the fibres to acquire electrical charge. After carding, needling of the non-woven fabric is performed in order to produce a felt product. The needling process also acts to enhance the charge on the web.

The Table shows the efficiency of filter materials described above. The efficiency is the percentage of particles borne in an air flow passing through the filter material which are trapped by the filter material. The tests to determine the efficiencies of the filter materials were performed according to the British Standard BS 4400 test. A 2g mass of the material in question, in standard capsule form, was tested using sodium chloride particles of  $0.65\mu m$  mean diameter.

Blend	Efficiency/%
polypropylene/halogen free acrylic	99.25
polypropylene/PVC	99.0
polypropylene/halogen free acrylic/PVC	99.25

Thus all of the embodiments exhibit excellent filtering efficiencies.

Commercially produced filter materials made according to EP 0 246 811 utilise modacrylic as the halogen substituted polyolefine: indeed, modacrylic is the preferred fibre in EP 0 246 811. However, modacrylic suffers the disadvantage that, when incinerated, it produces highly toxic dioxins at unacceptably high concentrations. This is a considerable problem, because ISO 14000 standards, which cover a variety of environmental concerns, have been applied to the disposal of used air filtration materials. In some countries, notably in the Far East, legislation requires that such disposal is carried out by incineration. The polypropylene/halogen free acrylic blend described herein does not produce dioxins when incinerated, since halogen substituents are not present in the fibres. A further advantage is that acrylic fibres are cheaper than modacrylic fibres.

The polypropylene/PVC blend exhibits considerably improved performance over a period of time compared to the modacrylic filter material which represents the preferred embodiments of EP 0 246 811. The Figure shows % penetration curves 10, 12 over an extended time period for, respectively, the polypropylene/PVC blend and a polypropylene/modacrylic blend (the preferred embodiment of EP 0 246 811). The % penetration represents the percentage of air particles passing through the filter material in an air flow (and therefore the efficiency value = 100% minus the % penetration). The polypropylene /PVC blend stabilises at a penetration value of ca. 7.1%. In contrast, the polypropylene/modacrylic blend exhibits considerably higher penetration values, reaching ca. 9.5%. Furthermore, the performance of the polypropylene/modacrylic blend is not stable over time: rather, it appears to be increasing steadily. The results shown in the Figure indicate that filters made from the polypropylene/PVC blend will have excellent working lifetimes. The working lifetime is given by the time period in which a filter can perform to within a specified standard. The Figure, shows that if, for example, an application requires that the penetration is 8% or better, the polypropylene/modacrylic blend would only be suitable for ca. 224 days. In contrast, the polypropylene/PVC blend is still performing with this specification at the end of the present study, i.e. after 728 days, with the likelihood that this performance will be maintained for a considerably longer period of time.

Since PVC contains chloride substituents, it is possible that incineration of the polypropylene/PVC filter material will produce dioxins. It is not known whether any toxic substances produced by incineration of this material will be present in acceptable concentrations.

Blends comprising polypropylene with PVC and halogen free acrylic fibres are within the scope of the invention. The efficiency of this material is excellent, and the PVC component provides enhanced performance over an extended period of time. By

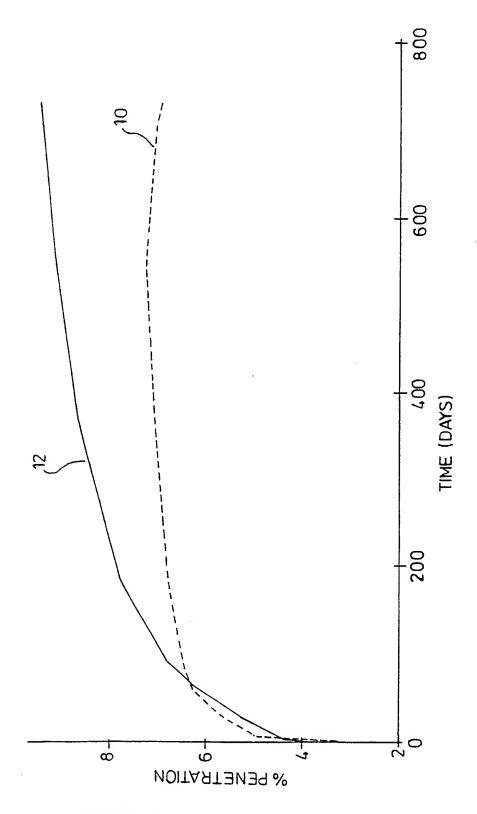
including a component of halogen free acrylic, the amount of PVC in the material is reduced and thus the concentration of any dioxins produced by incineration of the material is reduced.

In the examples described above, fibres of "standard" diameters, i.e., <u>ca.</u> 2-3 denier, were employed. The invention is not limited in this regard, and, in fact, it is likely that variation of fibre characteristics will result in variations in the characteristics of the filter material. "Coarse" denier fibres are, roughly speaking, of 5 denier and above, whilst fine denier fibres are, roughly speaking, 1.5 denier and below. Fibres of different coarseness can be blended. It should be noted that there are other factors than efficiency to consider, such as the pressure drop across the filter material, and the cost and/or weight of filter material required to obtain a desired performance characteristic. Variation of fibre coarseness is one way in which such factors might be optimised.

#### **CLAIMS**

- 1. An electrostatically charged filter material comprising a blend of polypropylene fibres and: i) halogen free acrylic fibres or ii) PVC fibres or iii) a mixture of halogen free acrylic fibres and PVC fibres.
- 2. A filter material according to claim 1 in which the halogen free acrylic fibres are produced by dry spinning.
- 3. A filter material according to claim 1 or claim 2 consisting of a blend of polypropylene fibres and halogen free acrylic fibres.
- 4. A filter material according to claim 1 or claim 2 consisting of a blend of polypropylene fibres and of PVC fibres.
- 5. A filter material according to claim 1 or claim 2 consisting of a blend of polypropylene fibres, halogen free acrylic fibres, and PVC fibres.
- 6. A filter material according to any of claims 1 to 3 or claim 5 in which the halogen free acrylic fibres comprise at least 40%, preferably at least 50%, more preferably at least 60%, most preferably at least 85% acrylonitrile.
- 7. A method for producing an electrostatically charged filter material comprising the step of blending together polypropylene fibres and: i) halogen free acrylic fibres or iii) PVC fibres or iii) a mixture of halogen free acrylic fibres and PVC fibres.
- 8. A method according to claim 7 in which lubricants and anti-static agents are substantially removed from the fibres before, during or after the blending of said fibres.

- 9. A method according to claim 7 or claim 8 comprising the step of carding the blend of fibres so as to form a non-woven fabric.
- 10. A method according to claim 9 in which the non-woven fabric is needled.
- 11. A method according to any of claims 7 to 11 in which halogen free acrylic fibres are produced by dry spinning.
- 12. A method according to any of claims 7 to 11 in which polypropylene fibres are blended with halogen free acrylic fibres but not with PVC fibres.
- 13. A method according to any of claims 7 to 10 in which polypropylene fibres are blended with PVC fibres but not with halogen free acrylic fibres.
- 14. A method according to any of claims 7 to 11 in which polypropylene fibres are blended with halogen free acrylic fibres and PVC fibres.



SUBSTITUTE SHEET (RULE 26)

#### INTERNATIONAL SEARCH REPORT

Interr ·al Application No PCT/GB 99/01990

A. CLASSIFI	CATION OF SUBJECT MATTER D04H1/42 B01D39/08 B03C3/28		
110 /	D04H1742 B01003700		
According to	international Patent Classification (IPC) or to both national classification ar	nd IPC	
B. FIELDS S	EARCHED	nhols)	
IPC 7	umentation searched (classification system followed by classification sym D04H B01D B03C		
	on searched other than minimum documentation to the extent that such do		ched
Electronic da	ta base consulted during the international search (name of data base and	d, where practical, search terms used)	
C DOCUME	ENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant	passages	Relevant to claim No.
X	EP 0 246 811 A (NAT RES DEV) 25 November 1987 (1987-11-25)		1,3,4, 6-10,12, 13
	the whole document		1.2
X	US 5 470 485 A (MORWEISER KARL-HEIN AL) 28 November 1995 (1995-11-28) the whole document	Z ET	1,3, 7-10,12
X	DATABASE WPI Section Ch, Week 199213 Derwent Publications Ltd., London, Class A88, AN 1992-099226 XP002119337 & HU 58 214 A (AGRONETT KORNYEZET), 28 February 1992 (1992-02-28) abstract		1,5,7,14
	-/-		
X Fu	inther documents are listed in the continuation of box C.	Y Patent family members are listed	in annex.
"A" docur corr "E" earlie fillin "L" docur whi cita "O" docur othe "P" docurate	ment defining the general state of the art which is not sidered to be of particular relevance or document but published on or after the international grate of the state of the international grate of the state of another that is cited to establish the publication date of another tion or other special reason (as specified) arment referring to an oral disclosure, use, exhibition or ar means arment published prior to the international filing date but or than the priority date daimed "& than the priority date daimed "& than the priority date daimed " and the state of the international search the state of the international search is not size of the state of the international search is not size of the state of the international search is not size of the state of the international search is not size of the state of the s	later document published after the into or priority date and not in conflict with cited to understand the principle or the invention.  document of particular relevance: the cannot be considered novel or cannot involve an inventive step when the discourage of particular relevance; the cannot be considered to involve an it document is combined with one or ments, such combination being obvious the art.  document member of the same pater.  Date of mailing of the international significant in the art.	claimed invention It be considered to ocument is taken alone claimed invention rentive step when the ore other such docu- ous to a person skilled
	19 October 1999	28/10/1999  Authorized officer	
Name a	nd mailing address of the ISA  European Patent Office, P.B. 5818 Patentlaan 2  NL - 2280 HV Rijswijk  Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  Fax: (+31-70) 340-3016	Polesak, H	

#### INTERNATIONAL SEARCH REPORT

Intern: 31 Application No PCT/GB 99/01990

		PC1/GB 99/01990		
C.(Continue	ation) DOCUMENTS CONSIDERED TO BE RELEVANT			
Category °	Citation of document, with indication, where appropriate of the relevant passages	Relevant to claim No.		
X	US 5 419 953 A (CHAPMAN RICK L) 30 May 1995 (1995-05-30) column 1, line 62-69 column 3, line 39-68	1,4, 7-10,14		
<b>X</b>	WO 97 40913 A (RACAL CORP CANADA INC) 6 November 1997 (1997-11-06) page 5, line 31,32	1,4,7,13		
		·		
	A210 (continuation of second sheet) (July 1992)			

## INTERNATIONAL SEARCH REPORT

..ormation on patent family members

Intern al Application No PCT/GB 99/01990

				1 .	
Patent document ited in search report		Publication date	Patent fam member(s		Publication date
EP 0246811	A	25-11-1987	GB 2190 HK 38	2764 A 0689 A,B 8195 A 8850 A	07-01-1993 25-11-1987 24-03-1995 17-01-1989
 US 5470485	A	28-11-1995	BR 950 CN 111 CZ 940 EP 067 ES 211 JP 271 JP 725 PL 30	7344 C 0387 A 2850 A 3341 A 4933 A 6512 T 6675 B 6024 A 17521 A	11-05-1995 24-10-1995 06-12-1995 14-02-1996 04-10-1995 16-07-1998 18-02-1998 09-10-1995 18-09-1995 08-02-1996
 НU 58214	A		NONE		
US 5419953	Α	30-05-1995	NONE		
WO 9740913	Α	06-11-1997	CA 22	98981 A 24857 A 48636 A	04-05-1999 06-11-1997 24-06-1999